PATENT SPECIFICATION

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COMPLETE SPECIFICATION

NO DRAWINGS

Detergent Briquettes

We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York 22, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to detergent briquettes, and provides a process for manufacturing such briquettes which are resistant to abrasion and accidental breakage when dry and are also readily disintegrable in water.

According to one aspect of the invention, a process for manufacturing a detergent briquette comprises mixing together normally solid water-soluble anionic synthetic organic detergent with a normally solid water-soluble inorganic salt, producing therefrom a particulate detergent composition containing up to 21% moisture, moistening the particles with 4 to 12% added water to increase the moisture content of the particles to 4 to 25%, pressing the detergent particles into a lightly compacted solid briquette at a pressure which is low enough to form a readily disintegrable briquette, and applying to the briquette a proportion between 1 and 5% by weight of the briquette of a readily water-soluble synthetic organic film-forming of a readily water-soluble synthetic organic film-forming polymer to form on the briquette surface a water-soluble film which is of strength sufficient to help make the detergent briquette resistant to abrasion and accidental breakage, when dry, and of such ready solubility that the detergent briquette will be readily disintegrable in water.

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The water soluble detergent briquettes obtained by the process according to this invention have been found to be commercially acceptable because of their improved resistance to abrasion and accidental breakage when dry, during packing, transport and handling, and also because they disintegrate and dissolve quickly in water. The word "briquette" as herein employed has no reference to shape but designates a solid made by compacting particles, preferably without crushing the particles.

According to another aspect of the invention, therefore, a water-soluble detergent briquette which is resistant to abrasion and accidental breakage, when dry, and which disintegrates readily in water, comprises a multiplicity of solid particles of water-soluble anionic synthetic organic detergent and water-soluble inorganic salt, of 4 to 25% moisture content, in the form of cohering particles initially of a moisture content of up to 21%, to the surface of which 4 to 12% water was added, lightly compacted and fused together at the moistened areas of contact by a low pressure to form a briquette, coated with an adhering and binding film of 1 to 5% by weight of the briquette of a readily water-soluble synthetic organic film-forming polymer of strength sufficient to help make the detergent briquette resistant to abrasion and accidental breakage, when dry, and of such ready solubility that the detergent briquette disintegrates readily in water.

Among compounds suitable for use as the normally solid water soluble anionic synthetic organic detergent are the sulphated and sulphonated synthetic detergents such as the alkyl aryl sulphonates, preferably alkyl benzene sulphonates of 12 to 20 carbon atoms in the alkyl group, e.g. propylene or other lower alkylene polymers of 12 to 15

carbon atoms; fatty alcohol sulphates, e.g. sulphates of higher fatty alcohols such as lauryl, myristyl and palmityl alcohols obtained from coconut oil; N-higher fatty acyl N-methyl tauride; alpha-sulphonated higher fatty acids; normal higher fatty alkyl sulphonates; and other sulphuric reaction product detergents having higher fatty alkyl or acyl hydrophobic radicals. In this specification the term "higher", when applied to alkyl and acyl groups, denotes a content of carbon atoms of 12 to 20. "Lower", indicates 1 to 4 carbon atoms in such radicals. All the above detergents are usually employed in the forms of their water-soluble salts, such as their alkali metal salts. Although sodium is the salt-forming cation most preferable, potassium and magnesium detergents may also be used, especially when employed as only a part of the total synthetic content of these briquettes.

The most preferable anionic detergents in the briquettes are the alkyl benzene sulphonates previously described, and these compounds, preferably in the form of the sodium salt of propylene tetramer benzene sulphonate, sodium salt of propylene pentamer benzene sulphonate and mixtures thereof, will most preferably constitute all or a substantial proportion, usually more than 50% of the synthetic anionic organic detergent content of the briquetted composition.

The addition of water-soluble inorganic salt, usually alkali metal salt, to the synthetic organic detergent helps to harden the organic detergent and makes it more suitable for formation into briquettes. Among the materials that are most suitable for this purpose are sodium tripolyphosphate, tetrasodium pyrophosphate, sodium sulphate, potassium sulphate and sodium chloride. Of the various salts that have been utilized, the water-soluble polyphosphates, such as the alkali metal polyphosphate salts, are highly preferred. These compounds, especially sodium tripolyphosphate and tetrasodium pyrophosphate, substantially improve the detersive ability of the synthetic organic detergent and have physical and chemical properties which aid in the production of a satisfactory form-retaining, yet readily disintegrable detergent briquette. Instead of the sodium polyphosphates the potassium salts may be used, usually in mixture with those of sodium.

The proportions of synthetic anionic organic detergent and inorganic builder salt may be varied within rather wide ranges so long as the mixture obtained is of acceptable cohesion while still disintegrating rapidly in water under washing conditions. Usually 20 to 40% and preferably 25 to 35% of anionic detergent such as sodium alkyl benzene sulphonate of the type described and usually 20 to 70%, preferably 30 to 55% inorganic builder salt, such as 40% of sodium tripolyphosphate, are employed. Various adjuvants such as perfume, pigment, dyes and other colouring agents, foaming agents, foam stabilizers, inorganic salt fillers, anti-redeposition agents, antibacterial compounds, sequestrants, anti-oxidants, corrosion inhibitors, anti-foams and other materials intended to improve one or more specific characteristic of the composition may also be added or included, usually in amount from 1 to 30%.

The balance of the detergent briquette composition, with the exception of a coating material to be described later, is water, present to the extent of from 4 to 25%, usually 8 to 19% and preferably 13 to 19%. Within the preferred range, briquettes containing 15% moisture are most usually acceptable. Relatively high proportions of moisture, such as these, are unusual in solid detergent products of this general type but, in conjunction with a thin coating of water-soluble film-forming polymer on the detergent briquette, have been found to be helpful in obtaining a product which is readily disintegrable in water.

A water-soluble polymer found to be especially effective in maintaining a high degree of water solubility of the detergent briquettes and simultaneously increasing their strength and resistance to abrasion is polyvinyl alcohol. The polyvinyl alcohols of commerce usually contain minor proportions of a lower fatty acid ester, polyvinyl acetate. This may be present in polyvinyl alcohols employed in this invention to the extent of about 10 to 30%. The useful polyvinyl alcohols ordinarily are also of a weight degree of polymerization between 30 and 300. Instead of the weight average degree of polymerization designation, such coatings may be characterised by viscosity measurements which are related to the degree of polymerization and molecular weight of the polyvinyl alcohol. Thus, a 4% solution of a polyvinyl alcohol of the mentioned degree of polymerization and polyvinyl acetate content will have a viscosity of about 1.5 to 7 centipoises at 20 degrees Centigrade. A good polyvinyl alcohol for use in accordance with this invention has a weight average degree of polymerization of about 50 to 250 (viscosity of about 2 to 6 centipoises) and a polyvinyl acetate content of 15 to 25%. A most preferred material has a weight average degree of polymerization of about 100 (viscosity of 3.5 centipoises) and polyvinyl acetate content of 20%. The polyvinyl acetate contents and average degrees of polymerization given are measurements of suitability of the polyvinyl alcohol for use as a briquette surface treatment. Above a polymerization degree of about 300

or acetate content over 30% the polyvinyl alcohol becomes difficult to dissolve quickly enough to permit rapid disintegration of the briquette. Below 10% acetate and below a polymerization degree of 30 the film may not be sufficiently protective of the briquette.

The polyvinyl alcohol used in this invention should be compatible with the detergent materials and various adjuvants that may be included with them. It should contain relatively little insoluble matter, should be colourless, odour-free and of neutral pH. Deviations from the above requirements may be made when warranted but in general a polyvinyl alcohol of the recited specifications would be most acceptable for household detergent products. Instead of the described polyvinyl alcohol, other readily water-soluble synthetic organic film-forming polymers of similar properties can be used, but usually such other compounds will not be as effective. However, polyvinylpyrollidone, sodium carboxymethyl cellulose, hydroxypropyl methyl cellulose and similar polymeric substances can produce water-soluble films for briquette surfaces. When used, it is preferred to mix them with polyvinyl alcohol so that the special advantages attending the presence of that material may be retained.

In making detergent briquettes it is possible to mix together the anionic synthetic organic detergent, such as sodium alkyl benzene sulphonate, together with the inorganic builder salts, such as sodium tripolyphosphate or other polyphosphates, and then briquette the physical mixture of these powders. Often, the cohesion of the materials is not accurately controllable and, if of satisfactory disintegration characteristics in water, the briquettes might also tend to break more readily when handled. A much more preferable way of producing these briquettes is by initially mixing the organic detergent and builder salt in an aqueous medium, such as a crutcher mix, in which a very uniform slurry of these components is produced, and then drying to desired form and moisture. The moisture content of the slurry should usually be from 30 to 50% and often is about 40%. The slurry will generally contain all the anionic organic detergent and inorganic builder salt to be found in the final briquette, together with the various adjuvants used, except such as may be unstable under the conditions of converting the slurry to a solid material. If desired, the materials of lesser stability, e.g., perfumes, may be added to the dried detergent prior to the application of moisture thereto and subsequent compacting.

The preferred method for converting the fluid mixture of briquette components to a most satisfactory form for compacting is by spraying the slurry through a small orifice or by otherwise atomizing it into a gas and passing it through a drying zone in which it is contacted and partially dehydrated by a heated drying gas. During the drying operation, detergent globules or beads are formed which become what may be considered to be the substantially spherical particles obtained from spray drying processes. Due to the expansion of the detergent globules when heated, often these beads are hollow and of low density, thereby facilitating their ready dissolution in water. The temperature of the drying gas used is usually between 300 degrees Fahrenheit and 600 degrees Fahrenheit, depending upon the type of material being dried. The detergent beads obtained contain a substantially homogeneous mixture of organic detergent and builder salt. The builder may be somewhat changed by the drying operation, it being known that sodium tripolyphosphate is partially converted to other phosphate salts when heated, but usually mixing and drying conditions are controlled to minimise such alterations. The dried beads are usually of apparent bulk density between 0.3 and 0.5 grams per cubic centimetre, preferably 0.4 grams per cubic centimetre, such density being measured by pouring a sample of beads into a cylindrical container of known volume and weighing the contents. Although the beads may be dried to a mosture content up to 21%, more often the drying is controlled so that 2 to 15% moisture is present in the beads when they are removed from the drying zone. Less than 2% initial moisture in the beads makes them more friable and also requires the addition of more moisture in a moistening operation before compacting. Such additional moisture may not be as readily absorbed as desired and can cause some adhesion of the detergent briquette to the pressing dies. Above 15% initial moisture, the detergent beads tend to become more adherent to other beads and equipment walls and become difficult to transport and handle easily, especially when warm. A preferred moisture content is 6 to 12% and for built alkyl benzene sulphonate detergents one usually finds that about 8% moisture is most desirable.

To facilitate production of a quickly dissolving briquette of desired density, the atomization of the slurry and the drying conditions should be controlled to produce detergent particles which are approximately globular and have diameters which will allow them to pass through a 5 mesh sieve and be retained on a 140 mesh sieve, preferably passing through an 8 mesh or 12 mesh sieve with less than 10% passing a 100 mesh sieve. The number of meshes indicates the number of openings per linear inch of

sieve or 1/2.5 times the number per centimetre. A small proportion of fines passing through the 140 mesh sieve may also be present without exerting a very detrimental effect on the product but efforts should be made to keep the content of such particles to a practical minimum. Beads that pass the above sieve test specifications will be substantially within the range of 0.1 to 4 millimetres diameter, preferable 0.15 to 2.5 millimetres. There are ranges of diameters over which the particles are distributed normally.

Detergent beads of the composition of the final product, except for the presence of the coating material, are next sprayed with additional water before compacting to briquette form. If desired and found useful, the water may contain dissolved or dispersed substances to regulate the spray properties or to be coated onto the particles or mixed therewith. However, usually it is preferred to employ only water, without a solute. Preferably the sprayed water is in finely divided droplet form and is directed onto the detergent beads as they are mixed or tumbled, so as to obtain a very uniform distribution of water spray througout the detergent. The amount of water sprayed onto the beads is 4 to 12%, preferably 4 to 10%, and most preferably about 7%. It will raise the moisture content of the beads to a range of proportions previously given. It has been found that it is usually preferable to add more moisture to the spray dried beads of initially lower moisture content than to beads originally somewhat higher in water content. The final moisture content of beads of the former type should also be in the lower parts of the ranges given. Thus, one might add 10% of moisture to detergent beads containing only 2% initial moisture and obtain a product initially very much like one resulting from moistening a 10% moisture bead with 5% of added water. However, for production efficiency, ease of handling and more ready absorption of applied moisture it is usually most desirable to have the moisture content of the spray dried beads above 6% and preferably about 8%.

During and after spraying the surfaces of the detergent beads with the required amount of moisture in finely atomized or fog from the beads are are mixed well together to assure that the moisture is evenly distributed and may be absorbed evenly by the particles. It is preferred to continue mixing for about 1 to 30 minutes, most preferably about 3 minutes, after spraying. The mixing should be by such a method that the particles are not substantially reduced in size. It has been found that tumbling allows maintenance of particle size within the ranges previously mentioned. If any particles form oversized agglomerates over 4 or 2.5 millimetres diameters they should be removed, preferably by screening. Although moistened detergent beads have been allowed to age before compacting, it is considered preferable to press these moistened particles into briquette form shortly after the application of additional moisture, usually within a couple of hours and preferably within about 30 minutes after moistening.

After the application of moisture the particles still are not objectionably adherent and can be readily conveyed to pressing means where they are lightly compacted into desired shape at a pressure low enough to form a readily disintegrable briquette, e.g. between 3 and 100 pounds per square inch, preferably between 15 and 50 pounds per square inch or 20 to 40 pounds per square inch. At such pressures the pre-moistened detergent beads are converted to form-retaining briquettes which, when coated with a material such as polyvinyl alcohol, are sufficiently strong for commercial handling but still disintegrate rapidly in water in home laundry washing machines. Such briquettes usually have an apparent density of 0.4 to 0.7 grams per cubic centimetre, preferably about 0.53 grams per cubic centimetre.

After pressing and before coating with polyvinyl alcohol or other suitable polymer, a form-retaining briquette is obtainable which can dissolve quickly in water and which still is of a surface strength and abrasion resistance that will allow it to be shipped without excessive breakage. However, it has been found that superior abrasion resistance is obtainable if the formed briquette is coated in accordance with this invention. In addition to the hydrating of the inorganic salts on the surface of the briquette, an aqueous solution of polyvinyl alcohol also forms a coating or film of that material around the briquette, giving it superior abrasion resistance and strength.

The polyvinyl alcohol coating, although it is water-soluble, appears to decrease the solubility rate of the briquette. According to the present invention, the pre-moistening of the detergent materials before compacting improves the solubility rate of coated briquettes and thereby permits the production of a readily disintegrable product which still has increased surface strength abrasion resistance. The application of moisture before compacting would normally but erroneously, be considered to cause an increase in cohesion of the particles, thereby making it even more difficult for them to be readily separated and dissolved by water. In addition to physically aiding in the production of a strong briquette the polyvinyl

alcohol also improves the washing ability of the detergent composition by increasing the degree of dispersion of dirt and other soiling substances removed from the materials being washed and preventing their redeposition upon the washed goods when the wash water is removed.

After the detergent briquette has been formed, it is coated with a polymer such as polyvinyl alcohol. This coating is preferably done by spraying onto the briquette surfaces a liquid comprising the polyvinyl alcohol. It is most preferred that the polyvinyl alcohol be dissolved in water and that the aqueous solution be sprayed or atomised in fine droplet form as evenly as practicable onto the surface of the briquette. The spray solution may contain glycerol or other low polyols to serve as plasticizers to help to keep the polyvinyl alcohol flexible and resilient, detergents or wetting agents to improve wetting of the briquette and neutralizing agent, e.g., sodium bicarbonate, to improve the odour of the polyvinyl alcohol by reacting with free acid present. Among other plasticizers that may find use are glycols, e.g., ethylene glycol and sugar alcohols such as sorbitol.

The amount of polyvinyl alcohol sprayed onto the briquette surface should be from 1 to 5% of the briquette weight and the moisture added from the solvent for the polyvinyl alcohol should be kept approximately within this range. A more preferred range of polyvinyl alcohol coating amounts is from 1.5 to 3% and about 1.9% of the coating is considered the best amount for general use. The polyvinyl alcohol is preferably applied as a 10 to 25% solution, such as a 17% solution, in water, the proportion of such solutions used being from 9 to 18%, preferably about 11%, of the briquette weight. When sprayed onto the detergent briquette surface, the polyvinyl alcohol and its solvent fill the voids between particles and also cover the surfaces of the particles. Because the briquette before coating is not perfectly smooth, it is evident that the coating will be thicker in some spots than in others but the average thickness is usually from 0.05 to 0.8 millimetre, preferably 0.08 to 0.5 millimetre, e.g., 0.2 millimetre. It has been noted that the dried film of polyvinyl alcohol, while is substantially covers the whole briquette, does sometimes contain minute thin spots or even perforations which might be of assistance in aiding the penetration of moisture into the briquette and speeding solution in water. Also sometimes air is entrained in the coating, increasing its thickness over the average thickness calculated from the weight of polymer applied and the area covered by it. Then too, the plasticizer, wetting agent, some moisture and other materials in the polymer spray tend to increase thickness and may promote solubility of the film.

After coating with polyvinyl alcohol solution, the briquette may be surface dried by forced air, heated air, infra-red rays or other suitable drying means to remove essentially all the moisture accompanying the polyvinyl alcohol in the coating spray. Thus, the final product will be of about the same moisture content as that of the particles before pressing. After drying, the briquette may be packed immediately in cartons ready for shipment and use. It is usually preferred that such cartons contain moisture barriers to assist in maintaining the correct moisture content in the briquette. Briquettes made in accordance with this invention may be shipped without breaking and even though stored for months before use will still disintegrate and dissolve rapidly when added to the tub of any of the conventional washing machines. They will also pass the rather severe strength and solubility requirements set for such products, withstanding a drop of at least 1 foot onto a hard surface, e.g., a metal plate, without breaking and also disintegrating in agitated water at 100 degrees Fahrenheit within a period of about one minute in a commercial washing machine of the top-loading type with a centre post agitator of usual operating and design characteristics.

The following examples illustrate the invention. All percentages and proportions in the examples, this specification and the appended claims are by weight.

EXAMPLE I

A crutcher mix was made of the following formula:

	%
Sodium tridecyl benzene sulphonate	19
Pentasodium tripolyphosphate	27.1
Sodium silicate (Na_2O/SiO_2 ratio of 0.43)	4.9
Sodium sulphate	12.2
Other adjuvants (anti-redeposition agents, fluorescent brightner, antioxidants)	1.1
Water	35.7
	100.0

The above crutcher formula was mixed to homogeneity at a temperature of about 165 degrees Fahrenheit for about 1/2 hour. It was then sprayed into heated drying gas at a temperature of about 500 degrees Fahrenheit and the sprayed fine droplets were partially dried as they passed through the heated gas. The spray dried detergent particles resulting were screened to pass through an 8 mesh sieve, hardly any particles being removed by the sieve, and it

was found that less than 10% of the particles were small enough to pass through a 100 mesh sieve. The spray dried detergent had a bulk density of about 0.4 grams/cubic centimetre, a moisture content of 8.5%, and a 1% solution in water was of pH 10. After cooling, the detergent was treated with a fine spray of water while being agitated by tumbling. The spray was applied for a short period, less than 5 minutes, after which tumbling was continued for about 10 to 15 minutes. The agitation was such that the detergent particles were not broken and were substantially within the 8 to 100 mesh range of the said particles. Agglomerates greater than 10 mesh were removed by screening. The moisture content of the resulting product was 14%. About five minutes after cessation of tumbling the detergent beads were compacted to briquettes, as follows.

At a pressure of 18 pounds per square inch the moistened detergent was pressed into briquettes which were approximately of cylindrical shape, 2½ inches in diameter and 1.31 inches thick and of density of 53 grams/cubic centimetre. The upper and lower faces of the briquettes were bevelled annularly and a diametric groove was formed across one face to facilitate division in half when so desired. The briquette weights averaged 53.9 grams.

Within an hour after briquetting the resulting tablets were sprayed with a solution of polyvinyl alcohol. This solution comprised 17% of a polyvinyl alcohol having a weight degree of polymerization of about 100 and a polyvinyl acetate content of about 20%, 1% sodium bicarbonate, 25% glycerine, 1.4% alkyl aryl sulphonate and the balance moisture. The polyvinyl alcohol solution was heated to a temperature of 150 degrees Fahrenheit and was sprayed onto the briquette in two applications, one for each major face of the briquette, resulting in the application of 1.9% polyvinyl alcohol and an average film thickness of about 0.4 millimetres, although it was apparent that the film was not perfectly even, containing many thin spots or perforations. The aqueous solvent in the coating solution was evaporated by infra-red heating. The briquettes resulting had a moisture content of about 14%. They passed tests for resistance to breakage and abrasion, being dropped from a height of one foot onto a hard surface without showing any damage. Tablets dropped from a height of about 5 feet did exhibit slight cracking. As to disintegration times, when placed in an operating washing machine of the top loading agitator type (5 gallon capacity) the briquettes disintegrated in 20 to 30 seconds. After this time there could not be observed any detergent briquette fragments, although not all of the detergent particles had completely dissolved. Wash tests run on briquettes containing surface coatings of polyvinyl alcohol of this type showed that they washed clothes whiter than the detergent from which the briquettes had been made.

EXAMPLE II

From a crutcher mix of about 40% moisture content, detergent beads of the following formula were made by spray drying by a method substantially like that of Example I.

	%
Sodium alkyl benzene sulphonate (the alkyl radical being a propylene polymer of 12 to 15 carbon atoms)	27
Sodium tripolyphosphate (includes phosphates produced therefrom during spray drying)	39
Sodium silicate	7
Sodium sulphate	17
Other adjuvants	1
Moisture	9
	100

The spray dried particulate detergent was substantially of spherical form, having a bulk density of about 0.4 and particle sizes ranging from 2.5 millimetres to 0.15 millimetres diameter, only about 5% thereof passing a 100 mesh sieve. Water was sprayed on the tumbling particles to raise the moisture content to 19%. After spraying, tumbling was continued for 5 to 10 minutes. Subsequently, the premoistened particles were lightly compacted at a pressure of about 15 to 20 pounds per square inch to a 40 gram tablet, 2¼ inches in diameter by 1 1/32 inch thick, of apparent density of 0.6 gram/cubic centimetre.

The briquette made was coated with 0.75 grams of polyvinyl alcohol of weight average degree of polymerization of between 50 and 250 and polyvinyl acetate content of 15 to 25%. The average coating thickness was about 0.2 millimetre after air drying. The polyvinyl alcohol was applied as an aqueous spray containing about 17% polyvinyl alcohol, 1% sodium bicarbonate, 1.5% wetting agent and 2.5% glycerine.

The briquettes made were sufficiently strong to withstand the drop test previously described, did not crumble and were not abraded severely during ordinary handling and disintegrated rapidly in use in commercial washing machines. Disintegration times of 20 to 33 seconds were obtained and re-checks made after storage for several months proved that disintegration times were still satisfactory.

EXAMPLE III

	%
Sodium higher alkyl benzene sulphonate (branched alkyl group of about 13 carbon atoms)	35
Sodium tripolyphosphate	38
Sodium silicate (principally orthosilicate)	7
Sodium sulphate	9
Other adjuvants	2
Moisture	9
	100

Detergent beads of the above formula were made by spray drying as described in the other examples. The beads were of particle sizes substantially between the sizes of the 8 and 100 mesh sieves and were of densities from 0.3 to 0.4 grams/cubic centimetre. A series of briquettes was made using different proportions of added moisture and various compacting pressures, resulting in products of different densities. It was established by such experiments that 4 to 10% added moisture and compacting at pressures of about 15 to 50, preferably 20 to 40 pounds/square inch, will result in coated briquettes (coated with about 2% of polyvinyl alcohol) which will disintegrate in agitated wash water within a minute and which will still be of sufficient strength, when dry, to withstand the drop test and will not be easily abraded during handling.

Thus, this detergent, at a density of 0.34 grams/cubic centimetre moistened with 5.6% water applied as a fine spray while the detergent was agitated, pressed at 25 pounds/square inch to a density of 0.53, produced a briquette which disintegrated in water within 25 seconds. When coated with 2% polyvinyl alcohol of the type described, this briquette would still disintegrate satisfactorily and was strong enough for commercial handling and shipment. Similarly, the addition of 9.1% moisture to beads of 0.33 grams/cubic centimetre and pressing at 18 pounds/square inch to a briquette of 0.53 grams/cubic centimetre or addition of 7.2% moisture to beads of 0.34 grams/cubic centimetre and pressing at 38 pounds/square inch to a density of 0.57 grams/cubic centimetre resulted in excellent, quickly disintegrating, strong briquettes, when subsequently coated with polyvinyl alcohol.

In contrast to the results of these experiments it was found that compressing beads of 9.2% moisture content and density of 0.37 grams/cubic centimetre at a pressure of 38 pounds/square inch to a density of even at little as 0.45 grams/cubic centimetre yielded a briquette that failed to disintegrate satisfactorily, even the uncoated briquettes of this type taking about 4 minutes to disintegrate in a washing machine.

WHAT WE CLAIM IS:—

1. A process for manufacturing a detergent briquette comprising mixing together normally solid water-soluble anionic synthetic organic detergent with a normally solid water-soluble inorganic salt, producing therefrom a particulate detergent composition containing up to 21% moisture, moistening the particles with 4 to 12% added water to increase the moisture content of the particles to 4 to 25%, pressing the detergent particles into a lightly compacted solid briquette at a pressure which is low enough to form a readily disintegrable briquette, and applying to the briquette a proportion between 1 and 5% by weight of the briquette of a readily water-soluble synthetic organic film-forming polymer to form on the briquette surface a water-soluble film which is of strength sufficient to help make the detergent briquette resistant to abrasion and accidental breakage, when dry, and of such readily solubility that the detergent briquette will be readily disintegrable in water.

2. A process as claimed in Claim 1 in which the detergent particles are pressed into a briquette at a pressure of 3 to 100 pounds per square inch.

3. A process as claimed in Claim 1 in which the readily water-soluble synthetic organic film-forming polymer is a polyvinyl alcohol of a weight average degree of polymerization of 30 to 300 and is of a polyvinyl acetate content of 10 to 30%.

4. A process as claimed in Claim 3 in which the polyvinyl alcohol is applied as a 10 to 25% by weight aqueous solution.

5. A process as claimed in Claim 3 or Claim 4 in which the polyvinyl alcohol forms on the surface of the briquette a coating of an average thickness of 0.05 to 0.8 millimetre.

6. A process as claimed in any of the preceding claims in which the anionic detergent is a non-soap alkyl aryl sulphonate detergent in which the alkyl group is of 12 to 20 carbon atoms, and the inorganic salt is a polyphosphate builder salt.

7. A process as claimed in any of the preceding claims in which the particulate detergent composition is made by mixing together, in an aqueous medium, a slurry of the anionic synthetic organic detergent and the inorganic salt, atomizing the slurried material into a drying gas and partially drying the atomized slurry to a particulate detergent of 2 to 15% moisture content, removing the detergent particles from the drying gas, applying 4 to 10% water to them as a fine spray while mixing the particles by tumbling so that the moisture is uniformly distributed over the surfaces thereof while the beads are maintained intact and substantially within 0.1 to 4 milli-

metres diameter range, the addition of water being such as to increase the moisture content of the beads to 8 to 19%, removing agglomerated particles over 4 millimetres diameter, and pressing to shape from the moistened particles a briquette of apparent density of 0.4 to 0.7 grams per cubic centimetre at a pressure between 15 and 50 pounds per square inch.

8. A process for manufacturing a detergent briquette substantially as described in the examples.

9. A water-soluble detergent briquette which is resistant to abrasion and accidental breakage, when dry, and which disintegrates readily in water, manufactured by a process as claimed in any of the preceding claims.

10. A water-soluble detergent briquette which is resistant to abrasion and accidental breakage, when dry, and which disintegrates readily in water, comprising a multiplicity of solid particles of water-soluble anionic synthetic organic detergent and water-soluble inorganic salt, of 4 to 25% moisture content, in the form of cohering particles initially of a moisture content of up to 21%, to the surfaces of which 4 to 12% water was added, lightly compacted and agglomerated together at the moistened areas of contact by a low pressure to form a briquette, coated with an adhering and binding film of 1 to 5% by weight of the briquette of a readily water-soluble synthetic organic film-forming polymer of strength sufficient to help make the detergent briquette resistant to abrasion and accidental breakage, when dry, and of such ready solubility that the detergent briquette disintegrates readily in water.

11. A water-soluble detergent briquette as claimed in Claim 9 in which the inorganic salt is a polyphosphate builder salt the briquette contains 8 to 19% moisture, is of an apparent density of 0.4 to 0.7 grams per cubic centimetre and is agglomerated to briquette form at a pressure between 3 and 100 pounds per square inch, and the film-forming polymer is a polyvinyl alcohol of a weight average degree of polymerization of 30 to 300 and polyvinyl acetate content of 10 to 30%, present in proportion of 1 to 5% of the briquette to form a coating on the briquette surface of average thickness of 0.05 to 0.8 millimetre.

12. A water-soluble detergent briquette as claimed in Claim 10 or Claim 11 comprising 20 to 40% anionic synthetic organic detergent, 20 to 70% inorganic builder salt, 8 to 19% moisture, 1.5 to 3% polyvinyl alcohol and 1 to 30% adjuvants, in the form of spray dried beads of particle sizes substantially within the 0.15 to 2.5 millimetre range, moistened and lightly agglomerated to a briquette which is subsequently coated with polyvinyl alcohol.

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